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**SAMPLING AND ANALYSIS PLAN**  
**CONFIRMATION SOIL SAMPLING OF 10-ACRE POND**  
**KERR-MCGEE CHEMICAL CORP. - SODA SPRINGS PLANT**  
**SUPERFUND SITE**  
**SODA SPRINGS, CARIBOU COUNTY IDAHO**

Prepared for:



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## LIST OF ACRONYMS

%D	Percent Deviations
%R	Percent Recovery
ATSDR	Agency for Toxic Substances and Disease Registry
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COCs	Contaminants of Concern
DEP	Massachusetts Department of Environmental Protection
DQI	Data Quality Indicator(s)
DQO	Data Quality Objective(s)
DU	Decision Unit
EDA	Exploratory Data Analysis
EDD	Electronic Data Deliverables
EPA	Environmental Protection Agency
EPH	Extractable Petroleum Hydrocarbon
F	Fahrenheit
FTL	Field Team Leader
GPS	Global Positioning System
HASP	Health and Safety Plan
IC	Institutional Control(s)
ICS	Interference Check Sample
IDEQ	Idaho Department of Environmental Quality
IDW	Investigation-Derived Waste
IS	Incremental Sample(s)
ISM	Incremental Sampling Methodology
KMCC	Kerr-McGee Chemical Corporation
LCS	Laboratory Control Sample(s)
MCL	Maximum Contaminant Level(s)
MDL	Method Detection Limit
MS	Matrix Spike(s)
MSD	Matrix Spike Duplicate(s)
Multistate Trust	Greenfield Environmental Multistate Trust, LLC, Trustee of the Multistate Environmental Response Trust
µg/L	Microgram Per Liter
mg/kg	Milligram Per Kilogram
NDs	Nondetects
NPL	National Priorities List
PC	Percent Complete
PM	Project Manager(s)
PPE	Personal Protective Equipment
PSLs	Project Screening Levels
QA/QC	Quality Assurance/Quality Control
RAWP	Removal Action Work Plan
RBPS	Risk-Based Groundwater Performance Standards
RI	Remedial Investigation
RLs	Reporting Limits
ROD	Record Of Decision

RPD	Relative Percent Difference
RSL	Regional Screening Level(s)
SAP	Sampling and Analysis Plan
Site	Kerr-McGee Chemical Corporation – Soda Springs Plant Superfund Site, Soda Springs, Caribou County, Idaho
SOP	Standard Operating Procedure(s)
SRI	Supplemental Remedial Investigation
SSL	Soil Screening Level
SU	Sampling Unit(s)
TBP	Tributyl Phosphate
TCRA	Time-Critical Removal Action
TPH	Total Petroleum Hydrocarbons
UCL	Upper Confidence Limit

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**1.0 INTRODUCTION**

This Sampling and Analysis Plan (SAP) specifies the methods and procedures to be used for collecting and analyzing soil samples during post-removal soil confirmation sampling of the Time-Critical Removal Action (TCRA) for the 10-Acre Pond at the Kerr-McGee Chemical Corporation (KMCC) – Soda Springs Plant Superfund Site, Soda Springs, Caribou County, Idaho (Site). This SAP was prepared on behalf of the Greenfield Environmental Multistate Trust, LLC, Trustee of the Multistate Environmental Response Trust (Multistate Trust), in support of ongoing environmental investigation and restoration activities at the Site. This SAP supports the Environmental Actions performed by the Multistate Trust as approved by and under the oversight of the U.S. Environmental Protection Agency (EPA), as Lead Agency for the Site, in consultation with the Idaho Department of Environmental Quality (IDEQ), as the Non-Lead Agency for the Site. The Multistate Trust's objectives are to ensure that the Site conditions are understood and appropriate actions are taken to protect human health and the environment. The TCRA will be performed by the Multistate Trust, and its contractors, in accordance with Section 104 of the Comprehensive Environmental Response, Compensation and Liability Act of 1980 (CERCLA).

**1.1 PROJECT ORGANIZATION**

Key project individuals for the post-removal 10-Acre Pond TCRA confirmation soil sampling and their responsibilities are listed below.

- Multistate Trust
  - Cindy Brooks: Managing Principal
  - Tasha Lewis: Portfolio/Program Manager
  - Lars Peterson: Project Manager
- EPA
  - Kathryn Cerise: Remedial Project Manager

- IDEQ
  - Dean Nygaard: Remedial Program Manager
  - Doug Tanner: Regional Project Manager
  - Stan Christensen: Site Project Manager
- Hydrometrics, Inc.
  - Mark Rhodes: Project Manager
  - Mark Walker: Project QA Manager
  - Caitlin Walter: Project Oversight

## **1.2 PROJECT SCHEDULE**

The schedule for planning document preparation, pond removal, confirmation sampling, data review, and preparation of final deliverables will conform to the following:

1. Implementation of the TCRA will be initiated within 45 days after EPA approval of the 10-Acre Pond Removal Action Work Plan (RAWP).
2. At this time, it is anticipated that pond removal and associated activities will be conducted from approximately June through November 2018.
3. Confirmation sampling will be conducted following the removal and prior to reclamation. At this time, confirmation sampling is anticipated to occur in September 2018.
4. Laboratory analysis and reporting is expected to be completed within 25 days after receiving samples, with the laboratory providing a Level 4 data package.
5. Data validation for confirmation sampling will be completed within 45 days of receipt of the final analytical data package from the laboratory.
6. A Draft Removal Action Completion Report will be submitted within 60 days of completing data validation.
7. A Final Removal Action Completion Report will be submitted within 30 days of receiving EPA's comments on the Draft Report.

## **1.3 DISTRIBUTION LIST**

A copy of the approved SAP for this investigation will be provided via email (electronic copy) and/or regular mail or hand delivery (compact disc or hard copy report) to the following individuals:

- Cindy Brooks (Multistate Trust);
- Tasha Lewis (Multistate Trust);
- Lars Peterson (Multistate Trust);
- Kathryn Cerise (EPA);
- Dean Nygard (IDEQ);



- Doug Tanner (IDEQ);
- Stan Christensen (IDEQ);
- Mark Rhodes (Hydrometrics);
- Mark Walker (Hydrometrics); and
- Caitlin Walter (Hydrometrics).

#### **1.4 SAMPLING AND ANALYSIS PLAN ORGANIZATION**

This SAP describes the procedures and tasks necessary to complete post-removal confirmation soil sampling of the 10-Acre Pond TCRA throughout the following sections:

- Background (2.0);
- Project Data Quality Objectives (3.0);
- Sampling Rationale (4.0);
- Method and Procedures (5.0);
- Data Evaluation (6.0);
- Corrective Action (7.0);
- Field Health and Safety Procedures (8.0); and
- References (9.0).

## **2.0 BACKGROUND**

KMCC operated a vanadium production facility at the Site from 1963 to 1999. As part of operations during this period, KMCC stored production process water and waste in several unlined ponds. From 1981 to 1989, significant uncontrolled releases of contaminated water occurred from the ponds to groundwater. On October, 4, 1989, EPA listed the Site on the National Priorities List (NPL) as a result of the releases.

### **2.1 SITE DESCRIPTION**

The Site is located in Caribou County, Idaho, approximately 1.5 miles north of Soda Springs (Figure 2-1). The approximately 538-acre Site is bordered by agricultural lands to the north, east, and south and by State Route 34 on the west. A phosphate processing plant (Monsanto facility) borders the west side of State Route 34, adjacent to the Site. With the exception of the Monsanto facility, the Site is bordered by rural areas within a 1-mile perimeter. The footprint of the proposed confirmation sampling at the 10-Acre Pond is shown in Figure 2-2.

The Site consists of six parcels of land, with 18 buildings currently remaining on-Site, many of which are not structurally sound or salvageable and are scheduled for demolition in 2018. Other than the remaining buildings, the 10-Acre Pond is the only remaining feature from the historical operations.

The Site elevation ranges 5,900 to 6,020 feet above sea level with average temperatures ranging from 29 to 55 degrees Fahrenheit (F). The wet season for the area is between March and June, with an annual average rainfall of approximately 15.9 inches. Wind rose data from Allen H. Tigert Airport (located about 2.5 miles south of the Site) show the predominant wind direction to the southeast.

There are six springs near the 10-Acre Pond that serve as the municipal water supply for the City of Soda Springs. One spring, Formation Spring, is located 1.5 miles northeast of the pond, and five springs are present in the Kelly Park area, located approximately 1.5 miles south of the 10-Acre Pond.

### **2.2 OPERATIONAL HISTORY**

KMCC (later Tronox Chemical Corporation) owned and operated the Site as a chemical manufacturing facility from 1963 through 2009. The facility produced vanadium from 1963 to 1999 and other secondary by-products such as fertilizer and cathode materials for rechargeable batteries in the later years of operation. Over the course of the operational history,

both lined and unlined ponds were incorporated for settlement, solvent extraction

### **FIGURE 2-1. LOCATION MAP**

## **FIGURE 2-2. 10-ACRE POND SAMPLING UNITS (SU)**

raffinate, tailings storage, product storage, and storm water retention. In 1997, KMCC constructed the 10-Acre Pond to contain liquids and solids from the closure of three on-Site ponds and from vanadium processing. In 2004, water and sediment from the closure of two additional 5-acre ponds were added to the 10-Acre Pond.

## **2.3 PREVIOUS INVESTIGATIONS AND REGULATORY HISTORY**

The timeline for investigations and regulatory history at the Site can be summarized as follows, with specific details provided in the 2017 Draft Phase I and Phase II Supplemental Remedial Investigation (SRI) Report (Tetra Tech, 2017), and in this RAWP:

- 1979: Initial discovery of environmental problems at the Site.
- 1981 and 1989: Significant failures in the ponds resulting in contaminated process water lost to vadose zone.
- 1985: Preliminary Assessment and Site Investigation performed by the State of Idaho Hazardous Materials Bureau, identifying the Site as a potential source of groundwater contamination.
- 1988: Site investigation under CERCLA.
- 1989: Site listed on the NPL.
- 1991 to 1994: Remedial Investigation (RI) under EPA oversight conducted, identifying groundwater as media of concern and the primary route of exposure as ingestion of groundwater. Identified contaminants of concern (COCs) include vanadium, molybdenum, manganese, tributyl phosphate (TBP), and total petroleum hydrocarbons (TPH) in excess of project screening levels (PSLs) (equivalent to the risk-based groundwater performance standards (RBPS) outlined in the Record of Decision (ROD) and arsenic in excess of the maximum contaminant level (MCL)). A secondary concern identified during the RI was potential ingestion and direct contact risk from elevated vanadium in material associated with the roaster reject area.
- 1995: ROD issued for non-operational portions of the Site with the following presented remedies:
  1. Eliminating uncontrolled liquid releases by replacing unlined ponds with lined ponds;
  2. Reuse and/or recycling of buried calcine tailings in fertilizer manufacturing at the Site over an 8-year period;
  3. Closure of select ponds and disposal of pond solids in an on-Site landfill;
  4. Capping of 'wind-blown' calcine and roaster reject material;
  5. Initiation of semi-annual groundwater monitoring program to monitor effectiveness of control measures in achieving groundwater PSLs for the COCs; and
  6. Incorporation of institutional controls (ICs) for off-Site exposure to groundwater for as long as PSLs are exceeded.

- 1997: Consent Decree implementing required remedy entered by the court targeting remedial actions for groundwater, roaster reject, and windblown calcine.
- 2000: ROD addendum changing reuse/recovery of calcine solids to capping after EPA concluded that the fertilizer plant could not meet ROD-specified timeframe.
- 2005/2006: KMCC divested and transfers Site to Tronox Chemical Corporation.
- 2009: Tronox filed for Chapter 11 bankruptcy.
- 2011: Bankruptcy court approved the Consent Decree and Environmental Settlement with the United States, 24 States, and Tronox resulting in the Site transfer to Multistate Trust.
- 2002, 2007, 2012, and 2017: Five-year reviews conducted by EPA. Results reflect that remedy for the Site conducted in accordance with ROD do not yet currently protect human health and the environment. Eight actions were identified in the 2012 review that needed to be taken at the Site, including:
  1. Delineate additional potential sources of Site-related COCs within former KMCC facility (noted as ongoing in the 2017 five-year review).
  2. Establish proprietary controls for the Site (noted as ongoing in the 2017 five-year review).
  3. Develop and implement IC plan governing groundwater use downgradient of the Site where COCs are known to exceed MCLs or RBPS (noted as ongoing in the 2017 five-year review).
  4. Investigate current and potential future uses and proximity of domestic wells in relation to the groundwater plume or plumes downgradient from the Site (noted as completed in the 2017 five-year review).
  5. Augment and expand groundwater well network to more fully delineate plumes (noted as ongoing in the 2017 five-year review).
  6. Conduct fence repair at Site to improve IC at landfill and calcine caps (noted as completed in the 2017 five-year review).
  7. Develop and implement a Site wide Operations and Maintenance Program (noted as completed in the 2017 five-year review).
  8. Conduct risk assessment of ecological receptors downgradient from the Site (noted as ongoing in the 2017 five-year review).

Recommendations in the 2017 five-year review include:

1. Finalize draft screening level assessment, identify data gaps, and complete characterization of source material in 10-Acre Pond.
  2. Develop and implement an IC Plan governing groundwater use at downgradient locations where COCs exceed MCLs or RBPS.
  3. Finalize multi-phased SRI.
- 2014: The Multistate Trust mitigated fugitive dust emissions at the 10-Acre Pond by regrading the pond beach area and applying dust and erosion control agents to the surface solids.

- 2015: The Multistate Trust initiated an SRI after receiving the Anadarko Litigation Settlement funds. The SRI is currently ongoing and has the following objectives:
  1. Investigate the possibility of additional sources of Site-related COCs and characterize if necessary;
  2. Augment and expand groundwater monitoring network;
  3. Improve delineation of molybdenum and vanadium contaminant plumes, local groundwater gradients, and anthropogenic influences on area-wide groundwater;
  4. Perform investigation to determine if Site-related COCs are impacting City of Soda Springs drinking water sources;
  5. Update Baseline Human Health and Ecological Risk Assessments by collecting new data; and
  6. Support Focused Feasibility Study by collecting new data.

## 2.4 ENVIRONMENTAL AND HUMAN IMPACT

The Site COCs molybdenum, vanadium, manganese, arsenic, TPH, and TBP have been identified at the Site by the ROD (EPA, 1995). It is currently unknown if the liner in the 10-Acre Pond has been compromised, and there is potential that the COCs have migrated to underlying soils and shallow groundwater.

Potential releases of COCs from the Site present a significant potential risk to both human and ecological receptors as aqueous concentrations in the 10-Acre Pond surface water exceed PSLs for Site groundwater by several orders of magnitude, with observed concentrations of up to 253,000 µg/L molybdenum (compared with a PSL of 180 µg/L); 32,000 µg/L vanadium (PSL of 260 µg/L); and 11,700 µg/L manganese (PSL of 180 µg/L). Potential release mechanisms at the pond include leaching and percolation/infiltration of COCs into underlying soil and shallow groundwater, discharge of COCs from shallow groundwater to surface water (downgradient springs), and windblown dispersion of COCs from seasonally dry areas within the pond area footprint off-Site.

Potential exposure pathways to both human and ecological receptors that have been identified include the following:

- Exposure of biological receptors to COCs in the 10-Acre Pond water and sediment;
- Exposure of biological receptors in springs/ponds/creeks impacted by off-Site COCs;
- Incidental ingestion and dermal contact of off-Site COCs in springs/ponds/creeks by recreational human receptors;
- Ingestion of fish that have bioaccumulated COCs in springs/ponds/creeks by recreational human receptors;

- Ingestion of waterfowl that have bioaccumulated COCs in springs/ponds/creeks by recreational human receptors;
- Ingestion of off-Site COCs in drinking water sources by human receptors; and
- Incidental ingestion and dermal contact of on-Site COCs in springs/ponds/creeks by trespassing human receptors.

Pursuant to Section 104 of the CERCLA Endangerment Determination, actual or threatened releases of hazardous substances from the 10-Acre Pond, if not addressed by implementing the proposed response action selected in the TCRA Action Memorandum (EPA, 2017a) and detailed in this RAWP, may present an imminent and substantial endangerment to public health or welfare, or the environment.



### **3.0 PROJECT DATA QUALITY OBJECTIVES**

The EPA Data Quality Objective (DQO) process is the agency-recommended planning process when environmental data will be used to derive an estimate of contamination (EPA, 2006). The recommended DQO process consists of seven steps that are used to develop performance and acceptance criteria that clarify study objectives, define the appropriate types of data, and specify tolerance of decision errors that will ultimately be used as the basis for establishing the quality and quantity of data needed to support decisions. These steps are outlined below for confirmation soil sampling proposed following the 10-Acre Pond TCRA for this Site, and are summarized in Table 3-1.

#### **3.1 STEP 1: STATE THE PROBLEM**

Historical operation of a vanadium production facility at the Site has resulted in consolidation of contaminated wastes in a 10-Acre Pond. The Site COCs that have been identified in the pond include arsenic, manganese, molybdenum, vanadium, diesel range TPH, and TBP. Contaminated surface soils around the property may leach into near surface soils and shallow groundwater or be blown to adjacent land on and off the Site property. Observations of tears in the 10-Acre Pond liner suggest that subsurface soil and potentially groundwater may be impacted by leaching from contaminated material within the pond. As part of the TCRA described in this RAWP, the 10-Acre Pond will be dewatered, dredged, the liner removed, and 1-foot of soil removed below the pond. The concentration and distribution of Site-related COCs in soil beneath the pond 10-Acre Pond is currently unknown. At the request of EPA and IDEQ, confirmation soil sampling will be conducted on the 10-Acre Pond TCRA footprint following the removal using relevant Incremental Sampling Methodology (ISM) guidance (ITRC, 2012) for sample collection, to allow accurate characterization of COC concentrations remaining in soils after the removal is completed.

#### **3.2 STEP 2: IDENTIFY THE GOALS OF THE STUDY**

Collect adequate data to characterize Site COC concentrations in surface (0-6 inches) and subsurface (6-12 inches) soils within the former pond footprint following removal of the 10-Acre Pond to allow comparison to applicable PSLs (Tetra Tech, 2017) and EPA Regional Screening Levels (RSLs) for (1) Composite Worker Soils (EPA, 2017b) and (2) Protection of Groundwater (EPA, 2017c).

**TABLE 3-1. 10-ACRE POND CONFIRMATION SOIL SAMPLING DATA  
QUALITY OBJECTIVES SUMMARY**

### **3.3 STEP 3: IDENTIFY INFORMATION INPUTS**

The information input needs related to the project goals include:

- The 10-Acre Pond TCRA footprint is considered a single decision unit (DU) for this investigation and has been divided into 20 sampling units (SUs), 10 of which are in the surface soil layer (0-6 inches) and 10 in a subsurface layer (6-12 inches).

Incremental samples (IS) for soil will be collected from each SU as described Sections 4.1 and 5.3 and will be analyzed for COCs as described in Section 5.8, to determine concentrations of COCs within each SU (i.e., spatial variability of COC concentrations beneath the 10-Acre Pond) and within the DU as a whole, for subsequent comparison to relevant standards (i.e., PSLs and EPA RSLs).

### **3.4 STEP 4: DEFINE THE BOUNDARIES OF THE STUDY**

Spatial boundaries of the study are defined based on the target populations of interest, which have been divided into 20 SUs within a single DU. The 10-Acre Pond post removal footprint has the potential to have been impacted by contamination of COCs stored in the pond. Considering that hot spots may be located in zones beneath areas where the liner has been compromised, the 10-Acre Pond has been divided into 1-acre plots, to help delineate smaller areas that may be elevated in Site COCs within the larger pond footprint. Thus, the overall DU (complete pond area) has been broken up into a 5 x 2 grid, with a surface layer (0-6 inches, 10 SUs) and a subsurface layer (6-12 inches, 10 additional SUs) within each 1-acre area (Figure 2-2). There is a possibility that bedrock will be exposed within the post-removal 10-Acre Pond TCRA excavation footprint. If an SU is more than 75 percent bedrock, then any surface soil within the SU will be included in adjacent SUs.

### **3.5 STEP 5: DEVELOP THE ANALYTIC APPROACH**

COC concentrations will be determined analytically for each of the ISs representing SUs within the 10-Acre Pond DU. Composite samples collected using ISM are designed to facilitate representative and accurate estimation of the average contaminant concentration within an SU or DU via a structured composite sampling and processing protocol, that reduces data variability and provides a reasonably unbiased estimate of mean contaminant concentrations (ITRC, 2012). The ISM approach includes recommendations for collecting a minimum of 30 subsamples of equal volume to obtain a representative composite sample within a given SU. ISM also recommends collecting three or more replicates (triplicates) within SUs to determine variability, and to aid in calculating statistics such as a 95% Upper Confidence Limit (UCL)

on the mean. Triplicate ISM samples will be collected from 50% of the proposed SUs to evaluate Site soil heterogeneity.

The EPA RSLs do not specifically target diesel range TPH, but instead targets low, medium, and high carbon content fraction ranges for both aliphatics and aromatics. Based on historic process use of No. 2 diesel, the TPH fractions of concern at the Site consist of diesel-range organics, however, EPA RSLs are specific to TPH carbon fractions (low C5-C8, medium C9-C18, and high C19-C32 aliphatics; low C6-C8, medium C9-C16, and high C17-C32 aromatics). Although fuel compositions may vary depending on the source of crude oil and the refining process, diesel range organics predominantly fall within the carbon ranges medium to high aliphatics (C9-C19+) and medium to high aromatics (C9-C12+), with minor fractions of lighter components (Brewer et al., 2013). The Agency for Toxic Substances and Disease Registry (ATSDR) notes that diesel fuels “predominantly contain a mixture of C10-C19 hydrocarbons” (ATSDR, 1999). In order to provide a general characterization of the diesel-range carbon fractions potentially present in soils, samples will be analyzed for Extractable Petroleum Hydrocarbon (EPH) fractions using the Massachusetts Department of Environmental Protection (DEP) EPH Method, which includes the C9-C18 aliphatics, C19-C36 aliphatics, and C11-C22 aromatics fractions. This approach will facilitate a relative comparison of TPH concentrations to EPA RSLs for the medium aliphatic (C9-C18 aliphatic), high aliphatic (C19-C32 aliphatic) medium aromatic (C9-C16 aromatic) and high aromatic (C17-C32 aromatic) carbon fractions.

Composite soil samples will be combined into a single sample container (i.e., sealed plastic bag) on Site, and shipped to the analytical laboratory for further processing and analysis of total arsenic, manganese, molybdenum, vanadium, EPH carbon fractions, and TBP. Laboratory processing and analysis is described in Section 5.8.

### **3.6 STEP 6: SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA**

#### **3.6.1 Confirmation Soil Sampling Performance Standards**

Accurate, precise, representative data are required for sample results to meet the project objectives of determining in-place post-removal soil concentrations to support future decision making. The post-removal soil concentrations within the 10-Acre Pond TCRA footprint will be compared to EPA RSLs for composite worker soil (EPA, 2017b) and protection of groundwater (EPA, 2017c). In addition, summary statistics may be calculated using soil concentrations for individual SUs and for the DU as a whole, including 95% UCL calculations or other statistics as appropriate. The RSLs and reporting limits (RLs) for soils at the Site are specified in Table 5-2. The laboratory RLs shall be equal to or less than the RSLs. If the RL exceeds the RSL, the Method Detection Limit (MDL) shall be equal to or less than the RSL, if

practicable. All laboratory analysis will be performed by Pace Analytical Laboratory. Data must meet the DQOs that have been specified for the parameters in this SAP.

### **3.6.2 Data Quality Indicators and Measurement Quality Objectives**

Quality Assurance/Quality Control (QA/QC) practices and analytical methods for this project have been designed to produce data of sufficient quality to support the intended data uses. Data quality will be assured by adherence to the methods and procedures outlined in this SAP, including full documentation of all field activities; specification and use of appropriate analytical methods; collection, analysis, and review of appropriate field and laboratory quality control samples; review of laboratory reports; verification and validation of analytical data (10 percent Level 4 and 90 percent Level 2a: see Section 6.1); and adequate design of the sampling plan. Each of these data quality indicator (DQI) elements is discussed in subsequent sections of this document.

Data assessment criteria for specific DQIs will be used to aid in the evaluation of overall data quality for data generated during the 10-Acre Pond TCRA. These DQIs are expressed in terms of precision, accuracy, representativeness, completeness, and comparability, which are described in detail below, and are summarized in the DQO summary table (Table 3-1).

#### **3.6.2.1 Precision**

Precision is defined as a measure of variability or reproducibility of replicate measurements, and is inversely related to the variability among the results obtained (e.g., highly variable results have low precision). Precision is assessed by field and laboratory duplicate result comparisons (including matrix spike duplicates). Field duplicates measure combined field and laboratory precision, whereas laboratory duplicates measure only laboratory precision.

Triplicate samples will be collected as part of the ISM protocol at 50% of the SUs sampled (5 of the 10 surface soil SUs, and 5 of the 10 subsurface soil SUs). The ISM triplicates measure the overall combined variability (precision) due to field heterogeneity and sampling techniques, and will be used primarily to evaluate Site soil variability, and in calculating statistics such as the 95% UCL on the mean as described in the ISM Guidance and in Section 3.5. Therefore, these ISM triplicates will not be used in the precision determinations described in this section. Instead, duplicates split from the bulk sample in the laboratory (described below in Section 5.4) serve as the duplicates for this sampling event.

Field and laboratory precision will be expressed in terms of relative percent difference (RPD) between the values resulting from duplicate analysis. RPD is calculated as follows:

$$RPD = [(x1 - x2)/X][100]$$

where:

- x1 = analyte concentration in the primary sample;
- x2 = analyte concentration in the duplicate sample; and
- X = average analyte concentration in the primary and duplicate sample.

The precision objective for soil sample duplicates will be agreement of sample results to within an RPD of  $\leq 35\%$  when both sample concentrations (original and duplicate) are greater than five times the reporting limit, and plus or minus two times the reporting limit when either of the sample concentrations is less than five times the reporting limit.

### **3.6.2.2 Accuracy**

Accuracy is a measure of the closeness of a reported concentration to a true (i.e., known to a certain degree of confidence) value. For this project, accuracy will be assessed by calculating percent recovery (%R) for laboratory control samples (LCSs), matrix spikes (MS), and matrix spike duplicates (MSD). Matrix spike %R indicates accuracy relevant to a unique sample matrix, while LCS %R indicates accuracy relevant to an analytical batch lot, and is strictly a measure of analytical accuracy conditions independent of samples and matrices. The %R of an analyte, and the resulting degree of accuracy expected for the analysis of QC spiked samples, are dependent upon the sample matrix, method of analysis, and the compound or

element being measured. The concentration of the analyte relative to the detection limit of the method also is a major factor affecting the accuracy of the measurement.

Accuracy is expressed as %R, calculated as follows for matrix spike (and matrix spike duplicate) samples:

$$\%R \text{ for matrix spike samples} = [(A-B)/C] \times 100$$

where:

- A = spiked sample concentration;
- B = measured sample concentration (without spike); and
- C = concentration of spike added.

The accuracy objective for matrix spike samples is recovery within the range of 75% to 125%, for samples where the spike value is at least four times the concentration in the original sample.

For LCSs, %R is calculated as follows:

$$\%R \text{ for LCSs} = [(SR/KV)] \times 100$$

where:

- SR = reported sample result for LCS; and
- KV = known value of LCS.

The accuracy objective for LCSs is recovery within the range of 80% to 100%, or within the 95% confidence limit of the known value. If method-specific control limits for spike recovery or LCS recovery for particular laboratory techniques differ from these, comparisons will be made with the method-specific control limits.

### **3.6.2.3 Completeness**

Completeness is defined as the number of samples or valid measurements obtained, compared with the total number planned, expressed as a percentage (“percent complete” or PC). PC is calculated as follows:

$$PC = NA/NI \times 100$$

where:

- NA = actual number of samples/valid measurements obtained; and
- NI = intended or planned number of samples/valid measurements.

For this project, two separate measures of completeness will be calculated, as follows:



1. Sampling Completeness – 40 separate ISs are planned for collection under this SAP (including ISM field triplicates). Field QC sampling frequencies are specified in Section 5.4. Sampling completeness will therefore be calculated as the actual number of samples collected (including both routine and QC samples) as a percentage of the planned number of samples included in this SAP. If bedrock expresses in the surface of an SU to an area greater than 75 percent of the total SU area, then that SU will not be sampled and the total number of samples will be modified before calculating sample completeness. Therefore, presence of bedrock will not adversely impact sampling completeness.
2. Data Completeness – Each IS will be analyzed for concentrations of COCs. Data completeness will be calculated as the actual number of valid (not rejected) individual measurements (parameter concentration values) obtained as a percentage of the planned number of total measurements. Results qualified during the data validation process (see Section 4.6.1) are counted as valid measurements, unless the value is rejected during the data verification/validation process.

The completeness goal for this project is 100 percent for sampling completeness, and 95 percent for data completeness. Sampling completeness will be assessed following field sampling activities, and data completeness will be assessed following data validation. If necessary, additional samples may be collected to ensure that project completeness goals are met.

#### **3.6.2.4 Comparability**

Comparability expresses the confidence with which data from one sample, sampling round, Site, laboratory, or project can be compared to those from another. Comparability during sampling is dependent upon sampling program design and time periods. Comparability during analysis is dependent upon analytical methods, detection limits, laboratories, units of measure, and sample preparation procedures.

Comparability is determined on a qualitative rather than quantitative basis. For this project, comparability of all data collected will be ensured by adherence to standard sample collection procedures, standard field measurement procedures, standard reporting methods, including consistent units, and the use of the same analytical laboratory. In addition, to support the comparability of the 10-Acre Pond TCRA confirmation sampling dataset with those obtained in previous investigations, all samples will be analyzed using EPA-approved methods; however, it should be noted that use of the requested ISM sampling protocols during this sampling event (as opposed to other protocols used during previous sampling) will inherently reduce the comparability of data collected under this SAP with previously collected data. All analytical standards will be traceable to standard reference materials. Instrument calibrations

will be performed in accordance with EPA method specifications, and will be checked at the frequency specified for the methods. The results of these analyses can then be compared with analyses by other laboratories and/or with analyses for other sites.

#### **3.6.2.5 Representativeness**

Representativeness expresses the extent to which collected data define actual environmental conditions. Similar to comparability, representativeness is determined on a qualitative rather than a quantitative basis. Sample collection, handling, and analytical procedures for the 10-Acre Pond TCRA confirmation sampling have been designed to maximize the representativeness of both sample collection and analytical results, within the fiscal,

logistical, and practical constraints typically encountered during environmental investigations. Collection of representative confirmation samples will be achieved by:

- Establishment of appropriate general sampling locations (Section 3.4);
- Collection of 30-point composite samples using methods compatible with the ISM (Sections 4.1 and 5.3); and
- Providing flexibility in designating specific sampling locations, to allow field crews and project managers (PMs) to adjust sampling locations if necessary to achieve a representative sample.

Generation of representative analytical data will be achieved by:

- Use of consistent sample preparation (i.e., homogenization and digestion), analytical, and reporting procedures by Pace Analytical Laboratory; and
- Analysis of specified laboratory QC samples to ensure analytical processes are in control.

Calculation of representative summary statistics (such as 95% UCLs or other statistics) for COCs will be achieved by:

- Validation of data to ensure data quality is sufficient for the intended use;
- Data evaluation to identify outliers and/or other non-representative analytical results; and
- Statistical characterization of datasets to determine the most appropriate statistical methods.

### **3.7 STEP 7: DEVELOP THE PLAN FOR OBTAINING DATA**

Surface and subsurface samples will be collected from the designated SU in the 10-Acre Pond DU using ISM procedures (ITRC, 2012). Samples will consist of composite samples (30 increments) collected in each of 20 SUs (10 surface and 10 subsurface SUs), along with field triplicates in 10 SUs (five surface and five subsurface SUs; see Figure 2-2). The exact number of SUs may be modified based on expression of bedrock in the 10-Acre Pond TCRA footprint, as some SUs may not have enough surface soil to sample. All IS subsamples will be collected using a systematic random grid approach. See Sections 4.1 and 5.3 for details on IS collection.

## 4.0 SAMPLING RATIONALE

Composite soil samples will be collected using ISM methodology from each of the SUs within the 10-Acre Pond DU. During Site preparation, described above, the field crew will conduct a visual survey of the SUs to determine the extent of bedrock surface expressions. At any SU with surface area consisting of 75 percent or greater bedrock, the remaining soil in that SU will be integrated into the nearest adjacent SU, at the discretion of the Field Team Leader (FTL). In SUs where bedrock expresses at the surface exists but is less than 75 percent of the SU area, the sample gridding will be modified to fit the smaller area, adjusting sample spacing accordingly to reach 30 samples evenly spaced throughout the area. The samples will be analyzed for arsenic, manganese, molybdenum, vanadium, EPH carbon fractions, and TBP.

### 4.1 INTEGRATED SAMPLING

An IS will be collected at each SU based on the recommendations in the ISM guidance (ITRC, 2012). Twenty one-acre SUs (ten surface and ten subsurface SUs) have been designated for IS to provide samples of the target populations of soil presented above. The SU size was selected to provide information on potential soil “hot spots” coinciding with areas where the pond liner has been compromised, as well as on the concentrations of COCs across the 10-Acre Pond area as a whole. Incremental samples will be collected using a 30-point IS method approach, ten SUs of which (five surface and five subsurface) will be sampled in triplicate to determine overall field and analytical variability, and aid in calculating summary statistics such as a 95% UCL on the mean, as necessary. The SU locations for these samples consist of a 2 by 5 grid within the 10-Acre Pond DU (Figure 2-2).

Within each SU, 30 increments will be collected using the “systematic random grid” approach (ITRC, 2012). This approach is designed to collect essentially random samples from within the DU while also providing that the samples are generally representative of the areal extent of each SU. Each 1-acre SU is broken up into a 3 by 10 grid, and a sample location selected randomly in one SU cell and systematically repeated through the entire SU. For non-contiguous SUs, for example if a SU has bedrock surface expressions less than 75 percent of the total area, a modified systematic random grid type layout will be developed, appropriate for the size, shape, and total area of the SU. Each incremental subsample location will be marked with labeled survey flagging or pin flags (corners and an approximate center for SUs with a rectangular shape and sufficient boundary points and the approximate center for more unusually shaped SUs when bedrock surface expressions are encountered) to facilitate surveying and to plot the SUs as sampled accurately on figures.

Mapping- or survey-grade global positioning system (GPS) instruments will be the primary method of identifying and marking sampling locations. If sample locations require modification due to presence of bedrock, adjusted locations will be documented using GPS.

Bedrock surface expressions will be documented by walking the bedrock perimeter while logging GPS points so that the bedrock areas can be added to Figure 2-2. The GPS measurement collected in the approximate center of the SUs will provide a single GPS point location for the SU database tracking.

The detailed sampling procedure is presented in Section 5.3.

## **5.0 METHOD AND PROCEDURES**

This section describes the procedures to be used to implement the 10-Acre Pond TCRA confirmation sampling, from field sampling and documentation, through laboratory analysis, data validation, and statistical evaluation of the dataset. Standard Operating Procedures (SOPs) relevant to this project are collected in Attachment B. It should be emphasized that SOPs are written to be general guidelines, and that the detailed procedures specified in this SAP supersede those specified in the SOPs. Sample documentation forms to be used during field sampling are in Attachment C.

### **5.1 FIELD EQUIPMENT**

Field sampling crews will take the following equipment (at a minimum) to the field:

- GPS Instrument (mapping- or survey-grade accuracy);
- 300-foot Field Measuring Tape;
- Pin Flags/Survey Tape;
- Soil coring device (manual and/or powered);
- Hammer drill;
- Shovel;
- Project Field Book;
- Field Forms Printed on Waterproof Paper;
- Indelible Ink Pens;
- Digital Camera;
- Disposable Plastic Trowels;
- 2-gallon resealable (Ziploc-type) plastic bags;
- Sample Coolers;
- Ice;
- 5-Gallon Buckets;
- Plastic Trash Bags;
- Paper Towels;
- Scrub Brush;
- Spray Bottles;
- Tap Water;
- Deionized/Distilled Water;
- Alconox or Equivalent non-Phosphate Detergent; and
- Nitrile Gloves.

## **5.2 CALIBRATION AND FIELD EQUIPMENT**

This investigation will not use field equipment requiring calibration. Normal maintenance for hand tools, cameras, or GPS units will be performed as necessary by the field team.

## **5.3 SURFACE SOIL SAMPLING**

### **5.3.1 Field Planning**

Before initiating the sampling event, the following preparatory activities must be completed:

- Schedule all sample analyses, sampling containers and preservatives, and sample delivery through Pace Analytical Laboratories.
- Obtain all necessary field equipment and supplies.
- Identify personnel to perform field sampling.
- Field team will review and discuss elements of the approved SAP and Health and Safety Plan (HASP). Personal protective equipment (PPE) and health and safety guidelines are specified for each activity in the HASP (Attachment D).

### **5.3.2 Field Planning Meetings**

Prior to the field mobilization, each field team member will review all project plans and participate in a field planning meeting. The meeting will be conducted by the PM and attended by all field staff and QA staff. All new field personnel will receive a comparable briefing if they do not attend the initial field planning meeting and/or the tailgate kick-off meeting. The meeting objective is to allow team members to become familiar with the history of the Site, special project requirements, and other items listed below. A field planning meeting may be held in the field instead of the office if this is more convenient for the personnel involved. Supplemental meetings may be conducted as required by any changes in Site conditions or to review field operational procedures.

The meeting will briefly discuss and clarify:

- Objectives of the fieldwork;
- Equipment and training needs;
- Health and safety requirements;
- Field operating procedures, schedules of events, communications, and individual assignments;
- Required QC measures; and
- Documents governing fieldwork that must be on Site.

A written agenda, reviewed by the PM, will be distributed and an attendance list signed. Copies of these documents will be maintained in the project files by the PM. Additional

meetings will be held when the documents governing fieldwork require it, when the scope of the assignment changes significantly, when the field personnel or Site conditions change, or if the PM determines that maintenance of QC protocol requirements merit another meeting.

### **5.3.3 Mobilization / Demobilization**

Mobilization will consist of property access scheduling, field personnel orientation, equipment and field supply ordering, staging, and transport to the Site. Equipment and field supply mobilization will include ordering, renting, and purchasing all equipment and supplies needed for the field activities. This will also include staging and transferring all equipment and supplies to and from the Site.

At the completion of the field activities, equipment and field supplies associated with the specific field activity will be demobilized as necessary from the Site. The FTL and/or designated field staff will be responsible for conducting an inventory of Site equipment and for ensuring that the equipment has been properly cleaned prior to removal off-Site and for securing any equipment to be left on Site. A final Site walkover will be performed to confirm the condition of the Site.

### **5.3.4 Investigation-Derived Waste**

Investigation-Derived Waste (IDW) may be generated during the project, consisting of excess soil sample material and decontamination water. Any excess soil from sample collection will be disposed of in the on-Site repository constructed as part of the 10-Acre Pond TCRA. All liquid wastes will be containerized and eventually shipped off with other liquid wastes from the TCRA.

### **5.3.5 Site Preparation**

Field staff will use GPS equipment to stake out the perimeter of each SU in the 10-Acre Pond TCRA footprint (Figure 2-2). The field team will then lay out the sample grid following the systematic random approach outlined in the ISM (Figure 2-2). If bedrock is present in the SU (up to 75 percent), the systematic random grid sampling scheme will be adjusted accordingly, to collect 30 samples evenly within the remaining soil area at the discretion of the FTL in consultation with the PM. If an SU is more than 75 percent bedrock, then any remaining soil within the SU will be included in adjacent SUs at the discretion of the FTL in consultation with the PM.

### **5.3.6 IS Sampling**

Sampling locations have been determined as discussed in Section 4.0. Exact sampling locations for each increment will be marked using a survey grade GPS, and properly documented as described below and in HSOP-2. In some instances, sampling locations may



need to be modified based on unknown underground structures or other considerations. Any significant changes in sample locations will be considered a variance and will need to be reviewed by the project team (including EPA/IDEQ) prior to sampling (HF-SOP-30).

Each increment will be collected from a depth of 0 to 6 inches for surface samples and from a depth of 6 to 12 inches for subsurface samples. An equal mass of approximately 100 grams of soil (volume of approximately 60 cubic centimeters) will be collected from each of the 30 subsample locations (the approximate volume collected by the coring device described below). The general procedure for collection of samples within a single SU is as follows:

1. Proceed to the flagged increment sampling location and prepare field documentation.
2. Remove large debris (i.e., stones) from the sample point location if necessary.
3. Collect surface (0-6 inches) increment using a stainless steel incremental sampling tool such as a step probe or soil coring device driven by a hammer drill. Note that alternative sampling tools may be used and documented as soil conditions warrant. If alternative tools are used, the mass of soil collected per subsample will remain consistent with the approximate mass/volume collected using the coring tool (100 grams or 60 cubic centimeters).
4. Eject the sample into a 2-gallon, heavy-duty, resealable plastic bag labeled with the appropriate surface soil sample number for the SU being sampled. Record description of soil characteristics.
5. Dig a small 0-6-inch pit in the location where the 0-6-inch increment was just collected, of sufficient dimension to allow placement of a probe or coring tool within the pit.
6. Collect the subsurface (6-12 inches) increment using a separate probe, soil coring device, or alternative sampling tool (not the same tool used for surface soil sampling).
7. Eject the sample into a 2-gallon, heavy-duty, resealable plastic bag labeled with the appropriate subsurface soil sample number for the SU being sampled. Record description of soil characteristics.
8. Backfill sample location.
9. Verify that all field documentation has been recorded.
10. Proceed to next flagged increment sampling location and repeat procedure until all 30 subsamples have been collected.
11. If triplicate field samples are being collected in the current SU, move to the next random starting location, and repeat procedure until three separate ISM samples have been collected in the SU.
12. Note that reused sampling equipment does not require decontamination between each increment, only between each SU or each triplicate sample. As described above, however, separate sampling tools should be used for surface and subsurface sampling.

13. After all 30 subsamples have been collected into the plastic bag, double bag and place in iced cooler to cool sample to at or below 6°C.
14. After completion of an SU, decontaminate sampling equipment as described in Section 5.5 and proceed to next SU.

Equipment/rinsate blanks will be collected twice during the sampling event in between SU composite samples. Equipment/rinsate blanks will be collected after the wash/rinse equipment cleaning cycle, by running laboratory grade DI water over and through the internal chamber of the coring device and/or alternative sampling tool, and then collecting the rinse water in a pre-preserved (nitric acid) plastic bottle for metals analysis only.

### **5.3.7 Sample Numbering**

The SUs delineated for sampling are shown on Figure 2-2, comprising 10 areas with a surface (0-6 inches) and subsurface (6-12 inches) sample to be collected in each area. SUs 1 through 10 are surface soil SUs, and SUs 11 through 20 are subsurface SUs. Surface soil SU 1 corresponds with subsurface soil SU 11, surface soil SU 2 corresponds with subsurface soil SU 12, and so on up to surface soil SU 10 corresponding with subsurface soil SU 20. Individual samples (including QC samples) will be assigned unique sample numbers according to the following sample numbering scheme:

AAAA-YYMM-XXXXX

where AAAA is a four-character code denoting the project (10AP for 10-Acre Pond), YYMM is a four-digit code denoting the year (i.e., 18 for 2018) and month (i.e., 09 for September) of collection, and XXXXX is a three- to five-digit code describing the specific SU sampled (SU1 through SU20) and the field QC identity, if required. Field triplicate samples will have A, B, and C appended to the SU descriptor (i.e., if triplicates are collected at SU5 the three samples will be designated SU5A, SU5B, SU5C).

Additional information to be included on the sample container label will include the date and time of collection and any sample preservation information.

### **5.3.8 Field Data QA/QC Procedures**

Field records will be checked for completeness at the end of each day of sampling by the members of the field sampling team. The check of field record completeness will ensure that all requirements for field activities have been fulfilled, complete records exist for each field activity, and that the procedures specified in the SAP were implemented. Field documentation

will ensure sample integrity and provide sufficient technical information to recreate each field event.

#### **5.4 FIELD QUALITY CONTROL SAMPLES**

For this investigation, 30-point composite samples will be collected in triplicate at 10 out of the 20 SUs (50%). The IS triplicates will be collected based on three separate random starting locations within the SU and will thus provide a measure of spatial heterogeneity; therefore, these samples will not be used for laboratory QA/QC purposes, but rather as a measure of Site soil variability as discussed in the ISM Guidance and in Section 3.5. Planned locations for collection of triplicate samples are shown on Figure 2-2; note, however, that some SUs may be entirely covered in bedrock and sampling not possible, and therefore some adjustment of SUs selected for triplicate sampling may be required. Triplicate sample containers will be assigned unique sample identification numbers, as described in Section 5.3.

Two equipment blanks will be collected over the course of the 10-Acre Pond sampling event. Equipment blank samples will be collected from the sample coring apparatus in between SU sample collection, after the equipment has been decontaminated (washed and rinsed).

A separate duplicate sample will be split from individual samples at the laboratory to be used for QA/QC at a frequency of 1 in 10 samples. Matrix spikes and matrix spike duplicates will be processed in the laboratory as splits from the bulk sample, and will also be analyzed in the laboratory at a frequency of 1 in 10. Separate field samples will not be submitted for duplicates and matrix spike/matrix spike duplicate analysis, as sufficient sample volume will be submitted to the laboratory to prepare the splits. Samples selected for duplicates will not be selected for matrix/spike/matrix spike duplicates. Duplicates and matrix spike/matrix spike duplicates will be randomly selected by laboratory personnel. Close coordination with the laboratory will be maintained to assure that they properly select samples for QA/QC upon sample arrival. Duplicate results will be used to assess the overall precision of the sampling and analytical methods employed. Matrix spike and matrix spike duplicates will be used to aid in determining precision and accuracy of laboratory procedures.

All field QC sample collection locations and procedures will be fully documented in the field notebook and on field sampling forms. Table 5-1 below summarizes the field quality control for this sampling event.

**TABLE 5-1. FIELD QUALITY CONTROL SUMMARY**

<b>Matrix</b>	<b>Analytical Group</b>	<b>Total # Samples</b>	<b>Field Duplicates/ IS Triplicates</b>	<b>Matrix Spike/Matrix Spike Duplicate</b>	<b>Rinsate Blanks<sup>(3)</sup></b>
Soil	As, Mg, Mo, V, EPH Fractions, TBP	Up to 40 <sup>1</sup>	1 per 10 samples for field duplicates (split in laboratory)  10 of the 20 SUs will be sampled as an IS triplicate <sup>2</sup>	1 per 10 samples for analysis	2 per event

- (1) Samples will not be collected for a particular SU if there is greater than 75 percent bedrock present. Ten of the twenty SUs will be sampled as an IS triplicate, each of which is considered an individual sample.
- (2) Triplicate IS samples will be collected from three different random starting locations within an SU (not split from a single sample). Triplicate samples will be used to evaluate soil heterogeneity.
- (3) For As, Mg, Mo, and V analysis only.

## **5.5 DECONTAMINATION PROCEDURES**

Decontamination procedures will be conducted for any sampling equipment that will be used at multiple SUs (i.e., coring devices, shovels). Decontamination fluids will be containerized on-Site and shipped off-Site with leachate and other liquid IDW generated from the 10-Acre Pond TCRA.

Equipment that is covered with large amounts of adhered soil should be pre-cleaned by brushing or scraping, prior to cleaning with fluids as described below. The following

sequential wash, scrubbing, and rinses will be carried out for each piece of equipment to be decontaminated (see HSOP-7, Attachment B):

- Non-phosphate detergent and tap water wash, using a brush if necessary.
- Tap water rinse.
- Deionized/distilled water rinse.

Equipment will either be allowed to air dry or will be dried with clean paper towels at the discretion of the field sampling crew.

## **5.6 SAMPLING DOCUMENTATION AND SHIPPING**

All samples collected and delivered to the laboratory for analysis will follow standard documentation, packing, and chain-of-custody procedures, as described in Attachment B (HSOP-4) and in the following sections.

### **5.6.1 Field Notes and Sampling Log Forms**

General procedures for documentation and labeling of samples, as well as recording information in field notebooks, are in HSOP-29 and HSOP-31 (Attachment B). All field-sampling activities will be recorded in bound, sequentially-paginated field log books and on

pre-printed field log sheets (Attachment C). All sample collection entries will include, at a minimum, the following information:

- Project name;
- Date and time;
- Sampling and other personnel present;
- Sample location;
- Sample ID number;
- Sample depth interval;
- Soil descriptions;
- GPS coordinates and datum;
- Weather observations;
- Any deviations from the SAP; and
- Other relevant project-specific Site or sample information.

Entries will be made in permanent ink, with corrections crossed out with a single line, dated and initialed. Field books will be signed at the bottom of each page by personnel making entries on that page. Completed field forms will also be signed by sampling personnel.

### **5.6.2 Photographs**

Photographs will be taken at the sampling locations and at other areas of interest on the Site or sampling area. They will serve to verify information entered in the field logbook. For each photograph taken, the following information will be written in the logbook or recorded in a separate field photography log:

- Time, date, location, and weather conditions;
- Description of the subject photographed; and
- Name of person taking the photograph.

### **5.6.3 GPS Coordinates**

Field teams will use pre-determined GPS coordinates of the sample location. GPS coordinates will also be recorded at the corners and at the approximate center of each SU (see Section 4.1). GPS coordinates will be collected at the perimeter of any areas of exposed bedrock within the 10-Acre Pond. The SOP for collection of GPS data (HSOP-5) is in Attachment B.

### **5.6.4 Sample Labeling, Packaging, and Shipping**

Each sample will be labeled with a unique sample identification number, according to Section 5.3.7. Samples will be stored in iced coolers or refrigerated following collection to maintain a sample temperature of less than or equal to 6°C. Samples will be shipped overnight to Pace

Analytical Laboratory, Billings, Montana for analysis, and will be delivered to the laboratory as soon as possible after acquisition, within one or two days after collection.

All sample shipments will be accompanied by the following documentation:

- Chain-of-custody form(s);
- Cover letter to the laboratory describing the accompanying samples; and
- Analytical parameter list with methods and required reporting limits (Table 5-2).

#### **5.6.5 Chain-of-Custody**

During and after sampling until the time of delivery, samples will be in the custody of sampling personnel or sample courier. Sample custody (responsibility for the integrity of samples and prevention of tampering) will be the responsibility of the field crews until samples are handed off to a designated courier. “Custody” refers to the samples being in the immediate care of the field personnel, either in physical possession, immediate view, locked up, or held in a secure area restricted to authorized personnel.

Chain-of-custody procedures will be followed to establish a written record of sample handling and movement between the sampling Site and the laboratory. All sample deliveries will be accompanied by a chain-of-custody record, to be signed by both the person delivering the samples and the receiving laboratory. Sampling personnel will retain one copy of the chain-of-custody after signing samples over to the laboratory, and the remaining copies will be transferred to the laboratory. Chain-of-custody forms will be provided by Pace Analytical Laboratory.

Upon arrival at the laboratory, sample custody shifts to laboratory personnel, who are responsible for tracking individual samples through login, analysis, and reporting. At the time of sample login, the laboratory will assign a unique laboratory sample number, which can be cross-referenced to the field sample number and used to track analytical results.

### **5.7 DATA MANAGEMENT**

In order to provide complete documentation of the 10-Acre Pond TCRA confirmation sampling, detailed field records will be collected by field personnel, as described in Section

**TABLE 5-2. ANALYTICAL METHODS, RSLs, AND REQUIRED REPORTING LIMITS FOR IS SAMPLES**

Parameter <sup>(1)</sup>	Digestion Method <sup>(2)</sup>	Analytical Method <sup>(2)</sup>	Required Reporting Limit (mg/kg) <sup>(3)</sup>	Method Detection Limit (mg/kg) <sup>(3)</sup>	Composite Worker Soil SSL (mg/kg) <sup>(4)</sup>	Residential Soil to Groundwater (mg/kg) <sup>(5)</sup>	Maximum Holding Time (days)	Preservation
Molybdenum (Mo)	3050B	6020	0.75	0.284	5,800	2	180	Cool to <6°C
Vanadium (V)	3050B	6020	0.75	0.042	5,800	86	180	Cool to <6°C
Manganese (Mn)	3050B	6020	0.25	0.0415	26,000	28	180	Cool to <6°C
Arsenic (As)	3050B	6020	0.5	0.257	3.0	0.0015	180	Cool to <6°C
EPH C9-C18 Aliphatics	DEP EPH 1.1	DEP EPH 1.1	10	1.01	440 <sup>6</sup>	1.5 <sup>6</sup>	7 (extraction) 40 (analysis)	Cool to <6°C
EPH C19-C36 Aliphatics			10	2.68	3,500,000 <sup>7</sup>	2,400 <sup>7</sup>	7 (extraction) 40 (analysis)	Cool to <6°C
EPH C11-C22 Aromatics			10	3.438	600 <sup>8</sup> 33,000 <sup>9</sup>	0.023 <sup>8</sup> 89 <sup>9</sup>	7 (extraction) 40 (analysis)	Cool to <6°C
TBP	8141A	8141A	33.3	11	260	0.025	14	Cool to <6°C

- (1) All parameters will be analyzed on bulk soil sample splits processed at the laboratory, following appropriate ISM sample conditioning (ITRC, 2012).
- (2) Laboratory analytical methods are described in the Laboratory SOPs in Attachment A.
- (3) The laboratory RLs shall be equal to or less than the RSLs. If the RL exceeds the RSL, the Method Detection Limit (MDL) shall be equal to or less than the RSL, if practicable.
- (4) EPA Regional Screening Levels (RSLs) for Composite Worker Soil (EPA, 2017b).
- (5) EPA RSLs for Protection of Groundwater Soil Screening Level (SSL) (EPA, 2017c).
- (6) RSL for medium aliphatics range (C9-C18).
- (7) RSL for high aliphatics range (C19-C32).
- (8) RSL for medium aromatics range (C9-C16).
- (9) RSL for high aromatics range (C17-C32).



5.6. All field notes, sample logs, and Site photographs will become part of the permanent data record, and will be included with the Final Removal Action Completion Report. All hard copy original field documents will be scanned to Adobe Portable Document Format (.pdf) to facilitate electronic storage and transfer. Documentation of sample custody (completed chain-of-custody forms) and sample transfer from sampling personnel to the laboratory, including cover letters and parameter lists with project-required analytical methods and reporting limits, will also be included as part of the field records package for this project.

Laboratory analytical reports and electronic data deliverables (EDDs) will be archived by the project contractor and transmitted to interested parties in electronic and/or hard copy formats. Summary data tables, statistical evaluations and calculations, data validation results and reports, draft and final project reports, and other files generated as part of the data analysis will also be archived by the project contractor.

## **5.8 LABORATORY ANALYSIS**

Appropriate laboratory processing is as important as correct implementation of field procedures in providing representative sample concentrations using the ISM technique. General procedures for preparing ISM samples in the laboratory are summarized in Section 6.2. of the ISM guidance (ITRC, 2012), including splitting the bulk sample prior to conducting separate preparations for different analytical techniques (metals, EPH fractions, and % moisture). Analytical considerations are presented in Section 6.3 of the ISM guidance. After the sample is split in the laboratory, the sample should be homogenized and subsequently subsampled with the same number of increments as were composited in the field (30 subsamples, in this case). To collect the subsamples, the sample is typically air-dried and some form of disaggregation and/or particle size reduction appropriate to the analysis being performed is conducted. A variety of techniques may be used for subsampling, but the final subsample mass must be used completely in the analytical sample preparation step. Section 6.2.2.7 of the ISM guidance (ITRC, 2012) describes the options for subsampling methods. Samples will be processed in accordance with Pace Analytical SOPs including Preparation of Soil Samples for Analysis by ICP and ICP-MS (EPA 3050B) and Metals Analysis by ICP-MS (EPA 6020) for metals, Determination of EPH (DEP EPH 1.1), and extraction and analysis of TBP (EPA 8141A), modified as appropriate to comply with ISM guidance. Each laboratory SOP is provided in Attachment A.

Quality control samples (duplicates, matrix spike, matrix spike duplicates) will be randomly assigned by laboratory personnel. Samples selected for use as a duplicate sample will not also be selected for use as a matrix spike/matrix spike duplicate, and vice versa.

All laboratory analysis will be conducted in accordance with the Pace Analytical Laboratories approved laboratory quality assurance plan (Attachment A). Laboratory analysis will consist

of determination of total metals concentrations, EPH carbon fractions, and TBP, using appropriate digestion and analytical procedures as outlined in Table 5-2. The laboratory will produce a Level 4 data package. Standard laboratory turnaround time (25 days for Level 4 reports) will be requested. Required project reporting limits and analytical methods for individual constituents are also presented in Table 5-2. The laboratory RLs shall be equal to or less than the RSLs. If the RL exceeds the RSL, the Method Detection Limit (MDL) shall be equal to or less than the RSL, if practicable.

## **5.9 DOCUMENTATION AND RECORDS**

All data generated from field activities such as field notebooks and sampling forms will remain in the possession of field sampling crews during the field sampling event. Upon return from the field, field sampling crews will duplicate and scan all hard copies of their records as soon as possible, to prevent the possibility of lost or misplaced information. Laboratory transmittal documents (chains-of-custody, cover letters, and analytical parameter lists) will also be duplicated and scanned after samples are delivered to the laboratory. Electronic and hard copies of these records will be stored in the project contractor's files and network system, and provided to EPA as part of the final project report.

## **6.0 DATA EVALUATION**

Data evaluation for the 10-Acre Pond TCRA confirmation sampling will consist of two components: (1) data verification and validation to assess data quality, and (2) statistical analysis of the validated dataset to determine the data distribution, summary statistics, and any other statistics (e.g., 95% UCL on the mean) necessary to fully characterize post-removal COC concentrations in soils. All data verification, validation, and statistical analysis will be conducted using standard office-type personal computers, configured with software appropriate for the proposed activities, including (at a minimum) Microsoft® Word and Excel, and the latest version of EPA's ProUCL software (Version 5.0 or later).

### **6.1 DATA VERIFICATION AND VALIDATION**

Overall completeness and adherence to project procedures will be assessed through validation and verification (HSOP-58, Attachment B). Verification includes confirmation of adherence to sample design, collection, handling, custody, transmittal, and documentation procedures. Validation includes the confirmation of adherence to specific analytical procedure criteria and protocols, and the assessment of data quality in terms of usability. Data validation and verification will be conducted by data management staff, with oversight by the PM.

The number and type of samples collected will be compared with project specifications to ensure conformance with the sampling process design. Review of sample collection and handling procedures will include verification of the following:

- Completeness of submittal packages;
- Completeness of field documentation, including chain-of-custody documentation;
- Field equipment calibration and maintenance and/or quality of field measurements; and
- Adherence to proper sample collection procedures.

The laboratory will provide the project contractor with EDDs. Upon receipt of EDDs, laboratory data reports will be reviewed for completeness and administrative errors. Following this procedure, data validation will be conducted by the project contractor in general accordance with the principles of the EPA national functional guidelines for inorganic data review (EPA, 2017d), USEPA National Functional Guidelines for Organic Superfund Methods Data Review (EPA, 2017e), IDEQ's Quality Management Plan (IDEQ, 2016), and as described in Section 6.2.5 of the approved Final Supplemental Remedial Investigation Work Plan (Tetra Tech, 2015). Consistent with the procedures described in Tetra Tech (2015), 10 percent of all data received from the laboratory as part of this investigation will be Level 4 validated and the other 90 percent will be Level 2a validated. The results of this validation will be summarized in the project report.

Level 2a validation includes evaluation and review of:

- When applicable, verification that the mass tuning of the instrument is performed at the proper frequency with required detection limits, along with continuous review of calibrations and method specific compliances;
- Results of laboratory method and field blanks, with associated appropriate qualifiers;
- QC sample %Rs and/or RPDs;
- Field sampling precision as evidenced by RPDs;
- Summary of method deviations; and
- Documentation supporting all qualifications.

Level 4 validation includes evaluation and review of:

- QC sample percent recoveries (%Rs), percent deviations (%Ds), and/or RPD to check if sample analysis falls within established QC limits;
- Additional QC checks may include: Interference check sample (ICS) %R, internal standard areas and/or %Rs, laboratory duplicate RPDs, and serial dilution %Ds. Results will be qualified accordingly if non-compliance to QC protocol is observed;
- Review of raw data including: sample quantitation reports, chromatographs, and/or mass spectra. Verification of detected results. Sample calculations for each analytical fraction reviewed in the data package and inclusion of the sample calculations in the data validation report; and
- All additional components of the Level 2a validation.

Qualifiers will be added to the data as required, and any data quality issues will be discussed in the validation report. The results of the confirmation sampling will be presented in the Final Removal Action Completion Report and will also include a discussion of any uncertainty or biases associated with the data, as indicated by field or lab QC sample results or other results of the validation. Validation results will be evaluated in terms of any potential effects on the intended data uses (characterization of SU and DU COC concentrations, calculation of summary and/or additional statistics), and the usability of all qualified data will be evaluated based upon how much uncertainty it introduces into the analysis.

Validated data will be incorporated into data summary tables in a format appropriate for both reporting and subsequent statistical analysis. Results included in summary data tables and used in statistical analyses will be verified for accuracy (against original laboratory reports/EDDs) prior to inclusion in the final project report.

### **6.1.1 Identification of Valid Samples**

The identification of valid samples involves interpretation and evaluation of the field records to identify problems affecting the representativeness of environmental samples. Judgments of sample validity will be documented in the validation report, any environmental data associated with poor or incorrect fieldwork will be identified, and the potential impacts on data utility (if any) will be noted in the data validation report.

## **6.2 STATISTICAL ANALYSIS**

Statistical analysis of the validated dataset obtained during the 10-Acre Pond TCRA confirmation sampling will be conducted using EPA's ProUCL software (Version 5.0 or later), supplemented by additional statistical software if necessary. For example, data may be used to calculate a statistically appropriate 95% UCL on the mean. Additional summary statistics and exploratory data analysis (EDA) may be conducted to support other project needs. The current version of ProUCL includes methods for distribution testing, calculating summary statistics, and calculating UCLs, among other methods.

Statistical analysis of soils data using ProUCL will likely include (but not necessarily be limited to):

- Summary statistics;
- Data distribution;
- Tests for outliers; and
- Calculation of UCLs.

The statistical approach for calculation of UCLs will be discussed with EPA/IDEQ after the datasets for constituents of interest have been characterized. The intent is to use all valid analytical data (including any nondetect data) in the statistical calculations. ProUCL allows for robust treatment of nondetects (NDs) by extrapolating estimated values for NDs using both regression on order statistics and Kaplan-Meier methods.

## **6.3 LABORATORY QUALITY CONTROL SAMPLES**

Laboratory QC sample types and frequencies (including duplicates, matrix spikes and matrix spike duplicates, and laboratory control standards) will be consistent with the quality assurance plan and SOPs of Pace Analytical Laboratory (Attachment A). The results of laboratory QC sample analysis will be reviewed during the data validation process.

## **7.0 CORRECTIVE ACTION**

Corrective action is the process of acting on non-conforming procedures that deviate from the procedures set forth in this SAP. Corrective actions can be implemented for field activities, laboratory analyses, data validation, and data assessment. The data validation report will document non-conforming conditions identified (e.g., when overall objectives for precision, accuracy, completeness, representativeness, or comparability are not satisfied), or when procedural practices or conditions differ from those described in this SAP. All corrective actions proposed and implemented will also be documented and will include measures to preclude a repetition of the original deficiency. The PM and QA Manager will be notified immediately of all non-conformances with this SAP that affect data quality before initiation of corrective actions both in the field and in the laboratory. Corrective action will only be implemented after review and approval by the PM and QA Manager. Approved corrective actions will be documented in the sampling project report.

### **7.1 FIELD CORRECTIVE ACTION**

Field conditions may vary from those anticipated in project planning documents; therefore, it may become necessary to implement minor modification to sampling procedures presented in this SAP (see HFSOP-30 in Attachment B). If such modifications are deemed necessary, EPA/IDEQ will be notified and verbal approval obtained prior to implementing the changes (if feasible), or as soon as possible thereafter. Any field modifications or corrective actions will be documented in the field logbook, and in the data validation and final project reports.

### **7.2 LABORATORY CORRECTIVE ACTION**

The laboratory is required to comply with the most recent version of their Quality Assurance Manual (February 2014), including all applicable SOPs. The Laboratory PM will be responsible for ensuring that appropriate corrective actions are initiated as required for conformance with this SAP. All laboratory personnel will be responsible for reporting problems that may compromise the quality of the data. The Laboratory PM or QA/QC Manager will be notified if any quality control sample exceeds the project-specified control limits. The analyst will identify and correct the anomaly before continuing with the sample analysis. A narrative describing the anomaly, the steps taken to identify and correct the anomaly and the treatment of the relevant sample batch (i.e., recalculation, reanalysis) will be submitted with the data package in the form of a cover letter.

### **7.3 DATA VALIDATION, VERIFICATION, AND ASSESSMENT CORRECTIVE ACTION**

Corrective action may be needed based on either the data validation/verification or data assessment (statistical evaluation). If corrective action is needed, recommendations will be made to EPA/IDEQ. Potential types of corrective action based on data validation / verification or data assessment may include re-sampling or re-analysis of samples by the laboratory. Recommended corrective actions may alter the project schedule, and will require approval by EPA/IDEQ prior to implementation. All corrective actions will be fully documented in the Final Removal Action Completion Report.

## **8.0 FIELD HEALTH AND SAFETY PROCEDURES**

A Site specific HASP has been prepared to cover all activities associated with the 10-Acre Pond TCRA and confirmation sampling and details Site safety concerns and protocol (Attachment D). PPE required for confirmation sampling includes the following Level D and Modified Level D PPE:

Level D PPE:

- Field clothing (long pants, short or long sleeve shirt);
- Hard hat;
- Steel-toed boots;
- Reflective vests; and
- Hearing protection (when needed).

Modified Level D PPE:

- Disposable Nitrile Gloves;
- Half-face respirator using HEPA filters (must be readily available at the Site for use during any activities that generate airborne dust);
- Tyvek coveralls (when needed); and
- Plastic boot covers (when needed).

Hearing protection, Tyvek coveralls, and plastic boot covers may not be needed during confirmation sampling; however, this equipment should be on Site in case field personnel identify a need for these PPE.



## 9.0 REFERENCES

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- EPA, 1995. Record of Decision. Kerr-McGee, Soda Springs. September 28, 1995.
- EPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process. EPA QA/G-4. EPA/240/B-06/001. Office of Environmental Information. February 2006.
- EPA, 2017a. Action Memorandum for a Time-Critical Removal Action for the 10-Acre Pond at the Kerr-McGee Chemical Corp. (KMCC) – Soda Springs Plant Superfund Site, Soda Springs, Caribou County, Idaho – Draft. November 2017.
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- EPA, 2017d. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Methods Data Review -- Final. EPA-540-R-2017-001. January 2017.
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- IDEQ, 2016. Quality Management Plan. December 2016.
- ITRC, 2012. Incremental Sampling Methodology. ISM-1. Washington, DC: Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team. February 2012.
- Tetra Tech, 2015. Final Supplemental Remedial Investigation Work Plan. Former Tronox/Kerr-McGee Chemical Corporation Superfund Site, Soda Springs, ID. October 2015.
- Tetra Tech, 2017. Draft Phase I and Phase II Supplemental Remedial Investigation Report. Former Tronox/Kerr-McGee Chemical Corporation Superfund Site, Soda Springs, ID. June 2017.

## **ATTACHMENT A**

### **PACE ANALYTICAL QUALITY CONTROL PLAN AND STANDARD OPERATING PROCEDURES**

**PACE ANALYTICAL QUALITY CONTROL PLAN  
AND STANDARD OPERATING PROCEDURES**

<b>SOP #</b>	<b>TITLE</b>
QA/QC Manual	Quality Assurance Manual: Quality Assurance/Quality Control Policies and Procedures
S-MT-O-001-Rev. 13	Determination of Extractable Petroleum Hydrocarbons (EPH)
S-MN-I-460-Rev. 19	Preparation of Solids Samples for Analysis by ICP and ICP-MS
S-MN-I-492-Rev. 28	Metals Analysis by ICP/MS
ALS-407	Organophosphorous Compounds by Gas Chromatography
ALS-617	Continuance Liquid/Liquid Extraction (CLE)

## **ATTACHMENT B**

### **HYDROMETRICS, INC. STANDARD OPERATING PROCEDURES**

**HYDROMETRICS, INC.**  
**STANDARD OPERATING PROCEDURES**

<b>SOP #</b>	<b>TITLE</b>
HSOP-2	DETERMINATION, IDENTIFICATION, AND DESCRIPTION OF FIELD SAMPLING SITES
HSOP-4	CHAIN-OF-CUSTODY PROCEDURES, PACKING, AND SHIPPING SAMPLES
HSOP-5	GLOBAL POSITIONING SYSTEM (GPS) EQUIPMENT OPERATION
HSOP-7	DECONTAMINATION OF SAMPLING EQUIPMENT
HSOP-29	LABELING AND DOCUMENTATION OF SAMPLES
HF-SOP-30	DECISION PROCESS FOR FIELD VARIANCE AND NONCONFORMANCES
HSOP-31	FIELD NOTEBOOKS
HSOP-58	GUIDELINES FOR QUALITY ASSURANCE OF ENVIRONMENTAL DATA COLLECTION ACTIVITIES – DATA QUALITY PLANNING, REVIEW, AND MANAGEMENT

## **ATTACHMENT C**

### **FIELD LOG SHEET**

## **ATTACHMENT D**

### **SITE HEALTH AND SAFETY PLAN**